BATCH EXTRACTION UNIT FOR TAR SAND PROCESSING STUDIES

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INTRODUCTION

In his early studies on the hot water method of separating bitumen from Athabasca tar sand, Clark (1-4) identified two components of tar sand, the fine solids content and bivalent ion concentration, as being important variables affecting the degree of separation. He also showed that the addition of inorganic bases was generally beneficial and that the amount of mechanical energy imparted to the tar sand slurry during processing was important.

Clark's studies were carried out in a laboratory scale separation plant which took a charge of approximately 8 kg of tar sand (1). Beaker extractions, which consisted essentially of stirring 0.2 kg tar sand with hot water in a beaker, were used for demonstration purposes(5).

In a later study, Seitzer (6) described a stirred reactor (capacity 0.2 kg of tar sand) which utilized a broad U-shaped paddle for slurrying and which with proper manipulation of variables could be made to simulate pilot plant extractions (7). Malmberg (8,9) used this reactor to further study the variables identified by Clark as well as others. Sepulveda utilized a commercial high shear stirred reactor for slurrying and a commercial flotation cell for the separation (10). One of the main objectives of these different groups was to find a parameter or parameters which would explain differences in bitumen yield from different tar sand samples under standard conditions, in short, the factors affecting tar sand processibility. Although much useful information was generated, this objective was not fulfilled.

This paper provides details of the design and operation of a convenient laboratory batch extraction unit, which was designed specifically for studying the chemical and physical properties of tar sand as they affect processibility. The unit was used to demonstrate the importance of surfactants in the hot water extraction process and to show the interrelationship between inorganic bases used as process aids and surfactants.

Description of the Unit

The objective was to design a unit that would a) give reproducible yield data; b) be sensitive to changes so that additives could readily be evaluated; c) be versatile, so that temperature, stirring rate, etc. could easily be changed; and d) establish trends that would parallel trends in larger continuous units.

One of the problems in dealing with tar sand is the natural variability of the deposit (7). It is therefore necessary to be able to homogenize a relatively large batch and use portions of this for comparative experiments yet the sample size must not be too small, otherwise loss of bitumen on the walls, etc. of the extraction apparatus will become significant. Based on these criteria, a sample size of 0.5 kg per experiment was chosen.

Details of the unit itself are given in Figures 1 to 3. The extraction cell is jacketed to provide for constant temperature during operation; the height is sufficient to provide a quiescent zone; and the cell is square to facilitate slurrying and subsequent agitation without the need for baffles. Air is added through the impeller shaft which allows good control even at low addition rates and avoids problems of plugging by fine solids experienced with other methods of addition, such as a porous plate. The air-introduction feature permits aeration of the slurry during mixing and allows one to simulate the secondary recovery step subsequent to primary recovery, practised in commercial units. After extraction is complete, tailings can easily be removed through a valve on the bottom of the cell. The valve also permits easy cleaning of the cell. The impeller speed is controlled by a variable speed fractional horsepower motor and is measured by a tachometer mounted on the impeller drive shaft. The impeller itself is easily removed so that different sizes and shapes can be used if desired.

The bitumen yield in the primary extraction step was known to be a function of stirring time and impeller speed for some types of tar sand (3,6,8). Experience with the unit has shown that for the times and speeds given in the procedure, the yield is relatively insensitive to these effects and errors resulting from small deviations are insignificant. When using a standard procedure the unit is ideally suited for studying processibility differences due to tar sand composition, the amount and type of process aid and other chemical additives.

Although a secondary recovery step similar to that practised in commercial units can readily be carried out, normally only primary bitumen recovery is used in processibility studies. The secondary recovery step is more efficient than commercial units and gives higher recoveries.

EXPERIMENTAL

Procedure I

Lab Scale Simulation of the Hot Water Extraction Process

Apparatus, Reagents, Materials:

1. Batch Extraction Unit and related components. The assembly is described in Figures 1, 2, and 3.

2. Constant Temperature Water Bath, maintained at 180°F (82°C). A pump allows water to be circulated from the bath to a jacket surrounding the stainless steel pot.

3. Timing Device, hot plate and distilled water.

Procedure:

1. Assure all air and water connections are sealed. Allow water from the bath to circulate through the surrounding jacket of the unit.

2. Heat approximately 1500 ml distilled water to 195°F (90.5°C) on a hot plate. Homogenize a quantity of the tar sand under test as outlined in Procedure II and submit three representative samples for bitumen/water/solids analysis (Procedure III). Collect 500 g of the remaining sample and weigh to the nearest 0.1 g.

3. Transfer 150 ml of the hot water to the stainless steel pot.
    Lower the motor and impeller assembly to its lowest possible
    position on the shaft rod and turn on. Allow the remaining
    water on the hot plate to cool to 180°F (82°C). (Impeller – ¼"
    from the bottom).

4. Add the 500 g tar sand sample to the pot. Within as little time
    as possible:
    a) Raise and lower the motor assambly to break up tar sand lumps
       present and leave the assembly two notches above the lowest one.
       (Impeller 3/4" above the bottom of the vessel).

    b) Adjust mixer to 600 rpm.

    c) Turn on air flow and adjust to approximately 465 cc/min on the
       flowmeter.

    d) Initiate timer.

    Mix in this manner for 10 minutes.

5. When complete, turn the air supply off and flood the mixture with
    approximately 1000 ml of 180°F (82°C) water, thus bringing the
    froth level to the top of the pot. Mix for an additional 10 minutes
    at 600 rpm. (No air).

6. Cease mixing and skim as much primary froth as possible from the
    top of the pot with the specially designed spatula. Collect the
    froth in a 4 oz sample bottle and submit for bitumen/water/solids
    analysis. (Procedure III).

7. For recovery of secondary froth mix the remaining material for
    5 minutes at 800 rpm while aerating at approximately 232 cc/min.
    Collect secondary froth in the same manner and submit for analysis.
    (Procedure III).

8. Drain the residual material from the bottom valve and wash the pot
    and impeller with toluene. When shut down, insure that the cir-
    culating water pump and air flow are turned off.

Comments

1. Residual material held in the stainless steel pot after recovery of
    the secondary froth may be differentiated as either secondary
    tailings or primary tailings in the following manner: Allow the
    mixture to settle one minute from the time mixing and aeration
    ceases. Decant the aqueous phase and denote as secondary tailings.
    The remaining heavier particles are termed primary tailings. Oil
    losses to the primary and secondary tailings may be obtained from
    bitumen/water/solids analyses of the samples.

Procedure II

Subdivision of Bulk Tar Sand Samples for Subsequent Analysis

Apparatus, Reagents, Materials:

1. Polyethylene sheet, approximately 3 feet square (1 m^2).

2. Spatula, with 12 inch (30.5 cm) stainless steel blade. A handle
    welded in place to the center of cutting edge facilitates use.

Procedure:

1. Empty bulk sample (ideal weight = 3000 g) onto the polyethylene sheet. Remove stones greater than ¼ inch (6.5 mm) in any dimension.

2. Chop the tar sand for a period of time ensuring all particles to be less than ¼ inch (6.5 mm) diameter. Pile and quarter the mixture.

3. Combine two opposite corners.

4. Repeat the chopping, quartering, and combining procedure as outlined, twice again. Resultant particles should not exceed 1/8 inch (3 mm) in any dimension.

5. Transfer approximately 50 g tar sand from the latter pile to a 4 oz sample jar and submit for analysis. Store the remaining tar sand in a tightly capped bottle at 40°F.

Procedure III

Determination of Bitumen/Water/Solids Content of Tar Sand, Froth, Middlings and Tailings Samples

Procedure:

The sample is separated into bitumen, water, and solids, by refluxing with toluene in a solids extraction apparatus (10). Condensed solvent and co-distilled water are continuously separated in a trap, the water being retained in the graduated section. The solvent recycles through the extraction thimble to further dissolve the bitumen. The bitumen/solvent and non-filterable solids fractions are subsequently separated by centrifuging. An aliquot of the resultant bitumen extract is evaporated to remove the solvent. The weight of bitumen water and solids is determined gravimetrically.

NaOH as a Process Aid

It was recognized very early in studies on the hot water extraction process that the acidity or alkalinity of the tar sand had an important effect on the efficiency of the separation (1). Strong inorganic bases, when added in small quantities, generally improved the separation efficiency. Separation efficiency was also thought to be dependent upon the concentration of certain substances which were present in the tar sand and dissolved or became suspended in the water phase during slurrying (2,3). Bowman (11) isolated some of the dissolved organics by foam fractionation technique and showed that the materials were surface active. Bitumen/water, air/water and air/bitumen interfacial tensions were measured and a thorough discussion of the theoretical implications of changes in interfacial transformations was presented. A similar discussion, from a thermodynamic approach was given by Leja and Bowman (12). The batch extraction unit described here was used to study the relationship between inorganic bases and surfactants in process streams.

A typical plot (8) showing the relationship between added NaOH and bitumen recovered in the primary separation step is shown in Figure 4. As expected, there is a corresponding increase in the pH of the aqueous tailings. Less obviously, as the amount of NaOH used increases, there is also a steady decrease in the surface tension of the secondary tailings and a corresponding increase in the organic carbon content of

the samples (Figure 5). This establishes a connection between surfact-
ants in the water and NaOH used during hot water extraction of bitumen
from tar sand. Further confirmation was obtained on titrating samples
of tailings. Titration curves are given in Figure 6. All tailings
samples exhibited an end point characteristic of sodium oleate soap.
Only at 0.056 wt% NaOH is there an end point which might be due to
NaOH.

These data show that NaOH neutralizes organic bitumen acids, to
give salts which are surface active in the aqueous phase. They further
show that NaOH results in increasing bitumen yield. To establish that
the important agent responsible for improved recovery is the surfactants
produced by caustic addition, tailings samples were centrifuged to
remove solids and concentrated by evaporation. The concentrate was
then added to the next experiment such that the organics in total
aqueous tailings from one run would be present in the slurry water of
a second run. The data is given in Table I. The surfactants recovered
from tailings are as effective as NaOH in improving recovery, showing
that surfactants are indeed the active process aid. With repetitive
recycle the effectiveness of concentrated tailings decreases, probably
due to losses in handling and through the formation of salts with
bi- and trivalent metals present in tailings streams. Table I also
shows that commercial surfactants can be used as well although these
are not economically competitive with NaOH at this time for commercial
use.

TABLE I

Effect of Surfactants on Bitumen Recoveries in
the Hot Water Extraction Process

Conditions		Bitumen Recoveries Primary Froth (%)
No Additive		61
NaOH: 0.04 wt% of T.S.		88
Concentrated, centrifuged tailings		87
No Additive		71
NaOH	0.008 wt% of T.S.	81
	0.024 wt% of T.S.	91
Tide	0.020 wt% of T.S.	82
	0.100 wt% of T.S.	89
Na Oleate	0.020 wt% of T.S.	70
	0.100 wt% of T.S.	83
Lignosulfonate	nil	35
HCX	0.020 wt% of T.S.	58
	0.100 wt% of T.S.	74

Conclusions

Details of the design and an operating procedure for a laboratory scale batch extraction unit have been described. Utilizing this unit, it has been shown that NaOH, when used as a process aid for tar sand processing, reacts with components of bitumen to form surfactants and that these surfactants are the primary agent responsible for improved bitumen recovery.
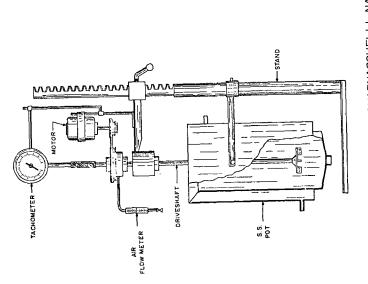
Acknowledgements

References

(1) Clark, K.A., and Pasternack, D.S., Ind. and Eng. Chem. 24(12) 1410(1932).

(2) Clark, K.A., The Can. Inst. of Mining and Metallurgy, Transactions, XLVII 257(1944).

(3) Clark, K.A., and Pasternack, D.S., Research Council of Alberta, Report No. 53, University of Alberta, Edmonton, Alberta (March, 1949).

(4) Clark, K.A. Can. Oil and Gas Industries, 3(6), 46(1950).

(5) Pasternack, D.S. Hodgson, G.W., and Clark, K.A. Proc. Ath. Oil Sands Conf. 200(1951).

(6) Seitzer, W.H., ACS San Francisco Meeting, April 2-5, 1968, p. F19.

(7) Innes, E.D., and Fear, J.V.D., Seventh World Petroleum Congress, Mexico City. Proceedings 3 633 (1967).

(8) Malmberg, E., and Bean, R.M., ACS San Francisco Meeting, April 2-5, 1968, p. F25.

(9) Malmberg, E.W., and Robinson, W.M., Ibid. p. F38.

(10) Sepulveda, J.E., Miller, J.D., and Oblad, A.G. PREPRINTS, Div. of Petrol. Chem. ACS 21 No. 6 110 (1976).

(11) Bowman, C.W., Proceedings of the Seventh World Petroleum Congress, 3 583 (1967).

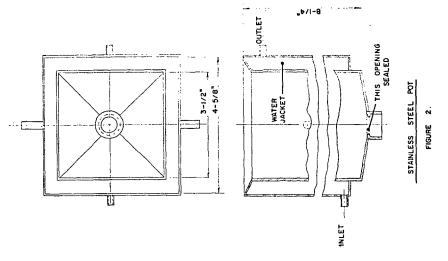(12) Leja, J., and Bowman, C., Can. J. Chem. Eng., 46(12) 479(1968).
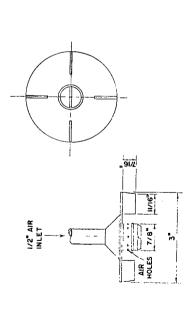
FIGURE 1

SYNCRUDE BATCH EXTRACTION UNIT

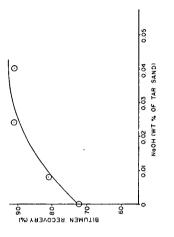TACHOMETER

MOTOR

AIR FLOW METER

DRIVESHAFT

S.S. POT

STAND

OUTLET

8-1/4"

WATER JACKET

THIS OPENING SEALED

STAINLESS STEEL POT

FIGURE 2.

INLET

3-1/2"

4-5/8"

BICHARD, BOWMAN, CYMBALISTY, GUARNASCHELLI, NAGY

FIGURE 4   The Effect of NaOH on Bitumen Recovery in
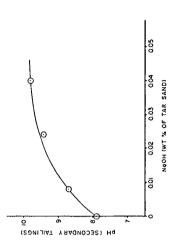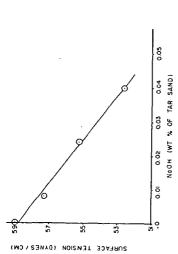the Primary Froth and pH of Secondary Tailings

IMPELLER

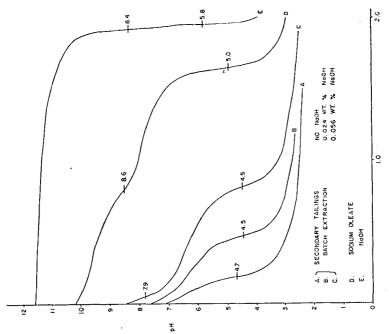FROTH RETRIEVAL SPATULA

FIGURE 3.

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FIGURE 6   Titration Curves for NaOH, Na Oleate and Aqueous Tailings

A. SECONDARY TAILINGS
B. } BATCH EXTRACTION
C.

D. SODIUM OLEATE
E. NaOH

NO NaOH
0.024 WT. % NaOH
0.056 WT. % NaOH

VOLUME OF TITRANT (ml) (0.1 NHCl)

pH

FIGURE 5   The Effect of NaOH on Surface Tension and Total Organic Carbon Concentration of Secondary Tailings

SURFACE TENSION (DYNES / CM)

TOTAL ORGANIC CARBON (ppm)

NaOH (WT % OF TAR SAND)

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